

FIG. 1

PROVIDE AN ALUMINA POWDER PRECURSOR

ADD ABOUT 1-10% MAGNESIA POWDER
PRECURSOR AND 1-10% TITANIA POWDER
PRECURSOR TO THE ALUMINA POWDER
PRECURSOR TO MAKE A GREEN POWDER
PRECURSOR

MIX THE GREEN POWDER PRECURSOR

PRESSING A GREEN BODY FROM THE GREEN
POWDER PRECURSOR

REMOVING RESIDUAL MOISTURE AND
ORGANIC MATERIAL FROM THE GREEN BODY

FIRING THE GREEN BODY IN AIR TO ABOUT
CONE 13

209030 0802500T

FIGURE 2

Some experimental data for a typical low-fired high alumina body

Alumina Content	Low Firing Alumina Composition
Acid Resistant	Silica-Free Formulation
Starting Grain Size	$\frac{1}{2} \mu\text{m}$
Grain Growth	1-3 μm (avg. 1.2 +/- 0.3 μm)
Material Preparation	Ball Milling, Spray Drying, Dry Pressing
Firing	Performed in Normal Atmosphere, Fiber-Lined Furnace, Fired to Cone 13
Specific Gravity	3.8 + (@ 1350°C)
Water Absorption	0
Thermal Conductivity @ 25°C (cal/cm ² /sec/°C)	0.05
Thermal Expansion @300 - 1000°C (°C)	8.3×10^{-6}
Thermal Expansion Rate	Substantially Uniform – There is Negligible Thermal Expansion Mismatch Between the Matrix and Second Phase
Toughness, K _{IC}	4-5 Mpa-m ^{1/2}
Rockwell Hardness (45N)	>80
Hardness (GPa)	16
Elastic Modulus	365 Gpa
Flexural Strength (kpsi)	47
Compressive Strength (kpsi)	>300
Tensile Strength (kpsi)	25
Dielectric Strength (v/mil)	250 (open atmosphere)
Dielectric Constant @ 1 kHz	8.2
Dissipation Factor @ 1 kHz	0.001
Loss Factor @ 1 kHz	.01
T _e (°C)	>900
Volume Resistivity @ 25°C	>10 ¹⁴
Surface Finish as Fired:	10 rms ± 2 (typical sample)
Ground:	5 rms ± 1 (typical sample)

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FIGURE 3

Long-Term Corrosion Study of Typical Low-Temperature High-Alumina (LTHA):

<u>MATERIAL</u>	<u>WEIGHT LOSS</u>
ZTA	-70 percent
SiC/SiC	-30 percent
LTHA Sample	-30 percent

**Materials Corrosion Test:
Independent Test**

Weight loss in mg/dm²/day

46.7% Hydrofluoric (HF) acid @ 25°C

	<u>5 day immersion</u>	<u>35 day immersion</u> (30 after 5)
SiC-Silica Free	1.00	1.00
ZrO ₂ -Toughened	1110.00	1070.00 ⁽¹⁾
Al ₂ O ₃ - 99.9%	1.92	2.26
LTHA Sample (Membrane-approx. 36% porosity)	1.0	0.16 ⁽²⁾
LTHA Sample (Solid)	1.64	0.09

NOTE: Weight loss is listed in mg/dm²/day rounded to nearest 0.01g.

- (1): Approximately 2/3 of the coupon was destroyed in 35 days of testing.
(2): This is a rather severe test in that the surface area is approx. 36% greater than the normal as tested.

Materials Corrosion Test:

50% H₃PO₄ @ 25°C

	<u>Cum. Mg/dm² (approx.)</u>	
	24 Hours	120 Hours
AD90	5.35	9.65
AD94	2.72	5.00
AD96	4.82	12.54
ADO96	5.61	11.59
AD99.5	6.75	10.26
TTZ	0.88	3.33
LTHA Sample	1.66	2.02



30% NaOH @ 25°C

Cum. Mg/dm² (approx.)

	24 Hours	120 Hours
AD90	24.98	51.15
AD94	15.24	32.27
AD96	2.13	6.10
ADO96	11.59	14.61
AD99.5	8.23	12.20
TTZ	0.61	0.61
LTHA Sample	1.72	2.01

NOTE: Weight loss is mg/dm²/day, rounded to nearest 0.01g.

Materials Corrosion Test:

Weight loss in mg/cm²/day

	<u>60% H₃PO₄ @ 60°C</u>	<u>30% NaOH @ 60°C</u>
A479 Al ₂ O ₃ (90%)	0.15	0.28
A479SS Al ₂ O ₃ (99.5%)	0.07	0.12
3NaI ₂ O ₃ (99.9%)	0.02	0.00
LTHA Sample	0.00	0.00

NOTE: Weight loss is mg/cm²/day, rounded to nearest 0.01g.

High Alumina Corrosion Test:

Independent Test

<u>CORROSIVE SOLUTION</u>	<u>HCl</u>	<u>HNO₃</u>	<u>H₂SO₄</u>
<u>MATERIAL</u>	<u>% WEIGHT LOSS</u>		
Product of Manufacturer A:			
85% Al ₂ O ₃	0.066	0.076	0.066
96% Al ₂ O ₃	0.081	0.087	0.200
LTHA Sample	(No Detectable Loss)		
Product of Manufacturer B:			
99.5% Al ₂ O ₃	0.217	0.163	0.216

PROCEDURES

1. Check the initial weight (approximately 5 grams)
2. Immerse into high concentration acid/base solutions
3. Dilute with 50 volume % of distilled water
4. Boil for an hour, and let soak overnight
5. Check the final weight
6. Calculate percent weight loss

$$\% \text{ LOSS} = (\text{INITIAL WEIGHT} - \text{FINAL WEIGHT}) / \text{INITIAL WEIGHT}$$

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FIGURE 4

Summary of Typical Low-Temperature High-Alumina Material Thermal Spray Features:

		<u>APPLICATION METHOD</u>	
		<u>Subsonic Plasma</u>	<u>HVOF</u>
Deposit Efficiency (DE)		85+%	75+%
Lay-Down Rate (surface speed = 1200 ipm)		4+mil/pass	1+mil/pass
Hardness (as applied)	Rockwell Vickers	90+ 1,100+	94+ 1300+
Surface Roughness (as applied)		200 – 300 μ in.	60 – 80 μ in.
Dielectric Strength		450+v/mil	950+v/mil
Bond Strength		10,000 – 12,000 psi	
Porosity (by optical microscopy)	Interconnected Surface		0% < 1/2 μ in.

Properties of LTHA Coating

		<u>APPLICATION METHOD</u>	
		<u>Subsonic Plasma*</u>	<u>HVOF**</u>
Deposit Efficiency (DE)		85.5% (82 - 91)	74% (65 - 83)
Lay-Down Rate (mil/pass) (surface speed = 1200 ipm)		3.5 (2 - 4.2)	0.98 (0.87 – 1.03)
Hardness (as applied)	Rockwell Std. Dev.	90.1 (88.9 – 91.7) 1.4	94.3 (94.1 – 94.7) 0.4
Surface Roughness (as applied)		200 – 300 μ in.	60 – 80 μ in.
Dielectric Strength		450+v/mil	950+v/mil
Bond Strength		10,000 – 12,000 psi	
Porosity (by optical microscopy)	Interconnected Surface		0% < 1/2 μ in.

* These averages are based on 132 sample coupons, randomly picked for testing out of approximately 30 spray runs containing 8 – 10 coupons per run.

** These averages are based on 30 sample coupons, randomly picked for testing out of approximately 10 spray runs containing 8 – 10 coupons per run.

() Indicates ranges of high and low values.

FIGURE 5

Tensile Test Data for 6061 Matrix Composites at 15v% (Test One)
 (NOTE: This test conducted with non-spherical grain)

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
6061 (avg.)	T - 6	9.9	40	45	10
LTHA Sample	T - 6	12.9	53	65	7
Tabular Al ₂ O ₃	T - 6	12.9	53	65	7

Tensile Test Data for 7093 Matrix Composites at 15v% (Test Two)

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
7093 (avg.)	T - 6	10.3	92	95.6	13.4
LTHA Sample	T - 6	13.1	80	85	0.9
Tabular Al ₂ O ₃	T - 6	13.4	58	67	4.2
B ₄ C	T - 6	14.6	84	98	2.6

Tensile Test Data for 7093 Matrix Composites at 10v% (Test Three)

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
7093 (avg.)	T - 6	10.3	92	95.6	13.4
LTHA Sample	T - 6	12.2	82.5	90.0	3.7
B ₄ C (96 samp.)	T - 6	13.2	89	96.7	4.1

Thermal Expansion Coefficient: 10 ppm/°F (Al = 13 ppm/°F)

Friction and Water Data:

Composite ID	Coefficient of Friction		Volume loss from Block (10 ⁻³ cu cm)
	Start	Finish	
20v% SiC/2124	.096	.119	6.34
25v% SiC/2124	.101	.123	6.23
30v% SiC/2124	.098	.119	4.15
20v% SiC/7091	.101	---	6.31
SPF-251 Std Coating	.141	.129	13.11

Test Results of 7093/Al₂O₃/xyp Composite

Sample	Vol. %	YS 0.2% (ksi)	UTS (%)	Elongation (Msi)	Modulus
Baseline T - 6	0	85.7	96.5	22.4	10.6
Tabular Al ₂ O ₃ T - 6	15	58.0	67.0	4.2	14.1
Medialox C25CR	10	25.1	33.9	2.43	---
Duralox DF500	10	36.5	48.7	7.56	---
Baikalo GE6	10	24.9	37.0	4.53	---
LTHA Sample Lot 1 T - 6	15	84.3	86.2	0.9	13.5
LTHA Sample Lot 2 T - 6	10	82.5	89.6	3.7	12.2

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